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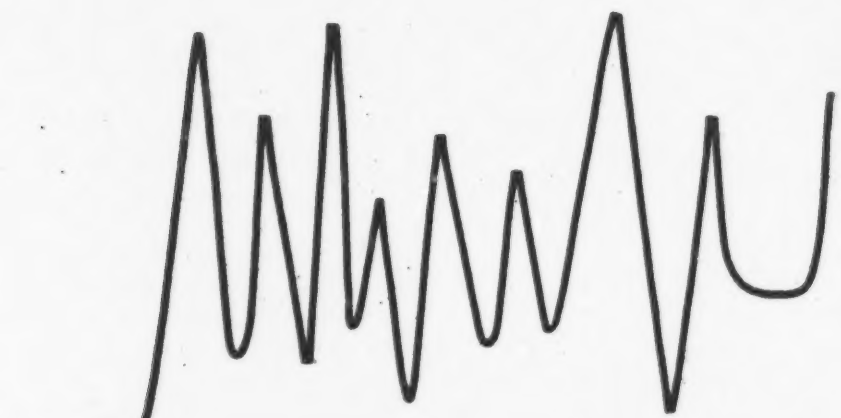
Test Methods for Evaluating Solid Waste

Physical/Chemical Methods



Technical Update

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16. Abstract (Limit: 200 words)

The Environmental Agency has issued Revision C to SW-846, "Test Methods for Evaluating Solid Waste" adding the following hexavalent chromium analytical methods to those approved for use in evaluating waste materials regulated under the Resource Conservation and Recovery Act: Method 8.545, Coprecipitation with Lead Sulfate; Method 8-546, Diphenylcarbazide Colorometric; and Method 8-547, Chelation - Extraction/Atomic absorption.

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TEST METHODS FOR EVALUATING SOLID WASTE

PHYSICAL/CHEMICAL METHODS

Technical Update

This manual (SW-846C) updates the Test Methods for Evaluating Solid Waste (SW-846), and was written by the Hazardous and Industrial Waste Division of the Office of Solid Waste.

U.S. ENVIRONMENTAL PROTECTION AGENCY
February 1982

Method 8.545
Chromium, Hexavalent
Coprecipitation with Lead Sulfate

1. Scope and Application

1.1 This method is to be used to determine the concentration of dissolved hexavalent chromium (Cr(VI)) in Extraction Procedure Toxicity characteristic (EP) extracts. This method may also be applicable to certain domestic and industrial wastes provided that no interfering substances are present.

1.2 The method may be used to analyze samples containing more than 5 ug of Cr(VI) per liter using either flame or furnace atomic absorption spectometry. In many cases, dilution of the extract will be necessary to achieve an optimal concentration range for Furnace Atomic Absorption spectometry. The dilution of the sample extract is sometimes desirable since it will reduce the likelihood of interferences from sulfate and chloride ions (see 4.1) which are present in many extracts and to increase the reproducibility of the method.

1.3 This method may not be applicable to certain types of samples and extracts. Extracts containing precipitate or cloudiness and those samples containing solid material must be pre-treated by digestion using the Basic Digestion Procedure in Method 8.548.

2. Summary of the Method

2.1 The method is based on the separation of Cr(VI)

from solution by coprecipitation of lead chromate with lead sulfate in a solution of acetic acid. After separation the supernate (containing Cr(III) is drawn off, the precipitate washed to remove occluded Cr(III), then the Cr(VI) is reduced and resolubilized in nitric acid and quantified as Cr(III) by either flame or furnace atomic absorption spectometry.

3. Sample Handling and Preservation

3.1 For guidance on sample handling and glassware cleaning procedures see Section 8.49.

3.2 Since the stability of Cr(VI) in EP extracts is not completely understood at this time, the analysis should be carried out as soon as possible.

3.3 To retard the chemical activity of Cr(VI), samples and extracts should be stored at 4°C until analyzed.

4. Interferences

4.1 Extracts containing either sulfate or chloride in concentrations above 1000 mg/liter should be diluted to a volume that lowers these interferences to below 1000 mg/liter before proceeding to analysis.

5. Instrument Parameters

5.1 Flame Atomic Absorption Spectometry

5.1.1 Fuel: Acetylene

5.1.2 Oxidant: Nitrous Oxide

5.1.3 Type of Flame: Fuel rich

5.1.4 Wavelength: 357.9 nm

5.1.5 Other operating parameters should be set as specified by the particular instrument manufacturer.

5.2 Furnace Atomic Absorption Spectrometry

5.2.1 Drying Time and Temperature: 30 sec at 125°C.

5.2.2 Ashing Time and Temperature: 30 sec at 1000°C.

5.2.3 Atomizing Time and Temperature: 10 sec at 2700°C.

5.2.4 Purge Gas Atmosphere: Argon

5.2.5 Wavelength: 357.9 nm

5.2.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

6. Special Apparatus

6.1 Glassware

6.1.1 Filtering flask, heavy wall, 1 liter capacity

6.1.2 Centrifuge tubes, heavy duty, conical, graduated, glass stoppered, 10 ml capacity

6.1.3 Pasteur pipets, borosilicate glass, 6.8 cm

6.2 Centrifuge: Any centrifuge capable of reaching 2000 rpm and accepting the centrifuge tubes described in 6.1.2 may be used.

6.3 pH Meter: A wide variety of instrumens are commercially available and suitable for this work.

6.4 Test Tube Mixer: Any mixer capable of imparting a thorough vortex is acceptable.

7. Reagents

7.1 Lead Nitrate Solution: Dissolve 33.1 grams of lead

nitrate, $Pb(NO_3)_2$ (analytical reagent grade), in deionized distilled water and dilute to 100 ml.

7.2 Ammonium Sulfate Solution: Dissolve 2.7 grams of ammonium sulfate, $(NH_4)_2SO_4$ (analytical reagent grade) in deionized distilled water and dilute to 100 ml.

7.3 Calcium Nitrate Solution: Dissolve 11.8 grams of calcium nitrate, $Ca(NO_3)_2.4H_2O$ (analytical reagent grade) in deionized distilled water and dilute to 100 ml. [1 ml = 20 mg Ca]

7.4 Nitric Acid, Concentrated, distilled reagent grade or spectrograde quality.

7.5 Acetic Acid, Glacial 10% (v/v): Dilute 10 ml glacial acetic acid CH_3COOH (ACS reagent grade), to 100 ml with deionized distilled water.

7.6 Ammonium Hydroxide, 10% (v/v): Dilute 10 ml concentrated ammonium hydroxide, NH_4OH (analytical reagent grade), to 100 ml with deionized distilled water.

7.7 Hydrogen Peroxide, 30%: ACS reagent grade.

7.8 Potassium Dichromate Standard Solution: Dissolve 28.285 grams of dried potassium dichromate, $K_2Cr_2O_7$ (analytical reagent grade), in deionized distilled water and dilute to 1 liter. [1 ml = 10 mg Cr]

7.9 Trivalent Chromium Working Stock Solution: To 50 ml of the potassium dichromate standard solution (7.8) add 1 ml of 30% H_2O_2 (7.7) and 1 ml concentrated

HNO_3 (7.4) and dilute to 100 ml with deionized distilled water. [1 ml = 5.0 mg Cr(III)] Prepare fresh monthly or as needed.

8. Calibration

8.1 Flame Atomic Absorption

8.1.1 At the time of analysis, prepare a blank and a series of at least four calibration standards from the Cr(III) working stock (7.9) that will adequately bracket the sample and cover a concentration range of 1 to 10 mg Cr/l. Add to the blank and each standard, before diluting to final volume, 1 ml 30% H_2O_2 (7.7), 5 ml concentrated HNO_3, (7.4), and 1 ml calcium nitrate solution (7.3) for each 100 ml of perepared solution. These calibration standards should be prepared fresh weekly, or as needed.

8.1.2 The use of simultaneous background correction is required for both calibration and sample analysis.

8.2 Furnace Atomic Absorption

8.2.1 At the time of analysis, prepare a blank and a series of at least four calibration standards from the Cr(III) working stock (7.9) that will adequately bracket the sample and cover a concentration range of 5 to 100 ug Cr/l. Add to the blank and each standard,

before diluting to final volume, 1 ml 30% H_2O_2 (7.7), 5 ml concentrated HNO_3 (7.4), and 1 ml calcium nitrate solution (7.3) for each 100 ml prepared solution. These calibration standards should be prepared fresh weekly, or as needed.

8.2.2 The listed instrument conditions (5.2) and calibration concentration range are for a Perkin-Elmer HGA-2100 based on the use of a 20 ul injection, continuous flow purge gas and non-pyrolytic graphite. The use of simultaneous background correction is required for both calibration and sample analysis.

9. Procedure

9.1 Transfer a 50 ml portion of the sample to a 100 ml Griffin beaker and adjust to a pH of 3.5±0.3 by dropwise adding volumes of 10% acetic acid record total volume of acid added. Proceed immediately to Step 9.2 taking no longer than 15 min. between these steps. Note: Care must be exercised not to take the pH below 3. If the pH is inadvertently lowered to <3, 10% NH_4OH (7.6) should be used to readjust the pH to 3.5 ± 0.3.

9.2 Pipet a 10 ml aliquot of the adjusted sample into (or an aliquot corresponding to 10 ml prior to pH adjustment) a centrifuge tube (6.1.2). Add 100 ul of the lead nitrate solution (7.1), stopper

the tube, mix the sample and allow to stand for 3 minutes.

9.3 After formation of the lead chromate, to help retain Cr(III) complex in solution, add 0.5 ml glacial acetic acid (7.5), stopper and mix.

9.4 To provide adequate lead sulfate for coprecipitation add 100 ul of ammonium sulfate solution (7.2), stopper and mix.

9.5 Place the stoppered centrifuge tube in the centrifuge, making sure that the tube is properly counterbalanced. Start the centrifuge and slowly increase the speed to 2000 rpm in small increments over a period of 5 minutes. Hold at 2000 rpm for 1 minute. Note: The speed of the centrifuge must be increased slowly to insure complete coprecipitation.

9.6 After centrifuging remove the tube and withdraw and discard the supernate using either the apparatus detailed in Figure 1 or careful decantation. If using the vacuum apparatus, the pasteur pipet is lowered into the tube and the supernate is sucked over into the filtering flask. With care the supernate can be withdrawn to within approximately 0.1 ml above the precipitate. Wash the precipitate with 5 ml distilled water and repeat steps 9.5 and 9.6 and proceed to 9.7.

9.7 To the remaining precipitate add 0.5 ml concentrated HNO_3 (7.4), 100 ul 30% H_2O_2 (7.7) and 100 ul

calcium nitrate solution (7.3). Stopper the tube and mix using a vortex mixer to disrupt the precipitate and solubilize the lead chromate. Dilute to 10 ml, mix and analyze in the same manner as the calibration standard (8.2).

10. Verification

10.1 For every sample matrix analyzed verification is required to verify that neither a reducing condition nor chemical interference affecting precipitation is present. This must be accomplished by analyzing a second 10 ml aliquot of the pH-adjusted filtrate that has been spiked with Cr(VI) (7.8). The amount of spike added should double the conentration found in the original aliquot. Under no circumstance should the increase be of less than 30 ug Cr(VI)/l for the furnace procedure. To verify the absence of an interference the spike recovery must be between 85% and 115%.

10.2 If the addition of the spike extends the concentration beyond the calibration curve, the analysis solution should be diluted with blank solution and the calculated results adjusted accordingly.

10.3 If the result of verification indicates a suppressive interference, the sample should be diluted and reanalyzed. If necessary use furnace atomic absorption to achieve additional sensitivity if required for the optimal concentration range.

10.4 If, after dilution of the sample, the interference persists an alternate method (chelation/extraction, or colorometric) should be used. Acidic extracts which yield recoveries of <85% should be retested to determine if the low spike recovery is due to the presence of residual reducing agent. This determination shall be made by first making and aliquot of the EP extract alkaline (pH 8.0 - 8.5) using 1N sodium hydroxide and then respiking and analyzing. If a spike recovery of 85-115% is obtained in the alkaline aliquot of an acidic extract that initially was found to contain <5 mg/l Cr(VI) one can conclude that the analytical method has been verified and the waste is not hazardous by reason of Cr(VI) EP Toxicity.

10.5 If none of the analytical methods approved for this analysis yield valid results the sample will be considered to exhibit the characteristic of EP toxicity unless exempted according to the provisions of 40 CFR 261.4(b)(6).

11. Analytical Notes

11.1 Nitrogen should not be used as a purge gas because of possible CN band interference.

11.2 The use of pyrolytic graphite should be avoided when possible, since in some situations an enhancement effect has been reported.

11.3 Pipet tips have been reported to be a possible

source of contamination.

11.4 The method of standard addition shall be used in accordance with general methods given section 8.49.

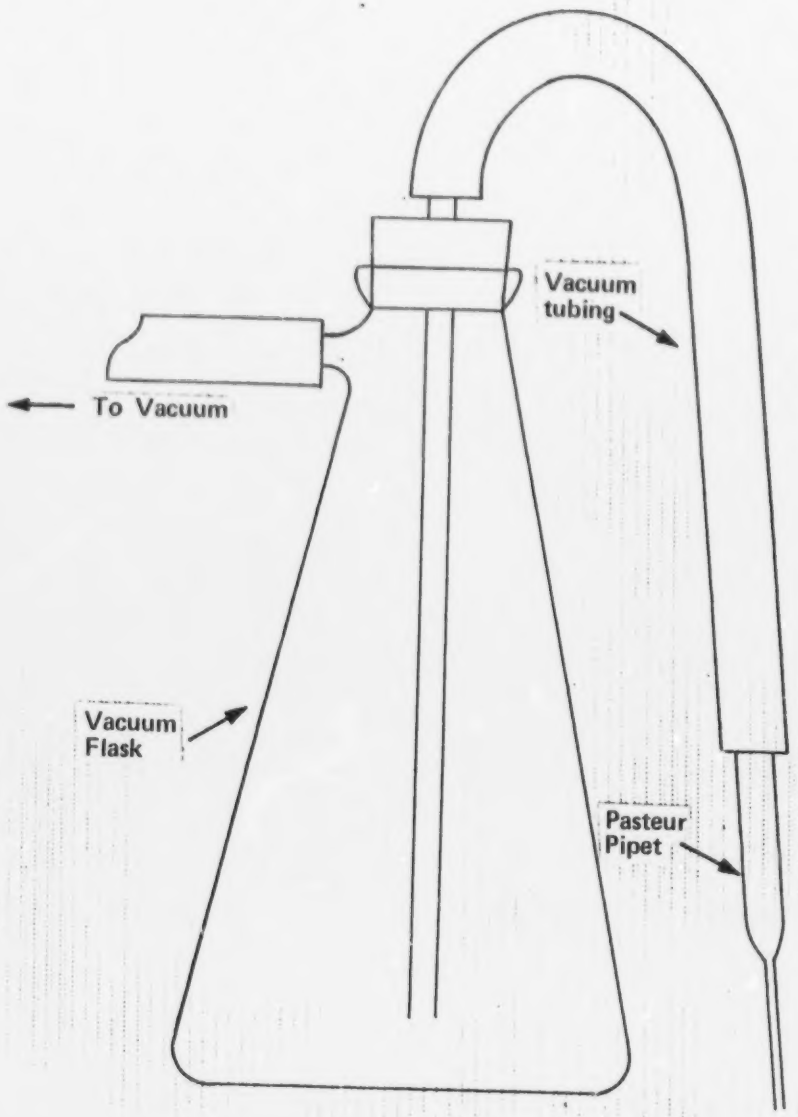

FIGURE 1

Method 8.546
Chromium, Hexavalent
Diphenylcarbazide Colorometric Method

1. Scope and Application

1.1 This method is to be used for the determination of dissolved hexavalent chromium Cr(VI) in Extraction Procedure Toxicity Characteristic (EP) extracts. The method may also be applicable to certain domestic and industrial wastes provided that no interfering substances are present. (See 4.1.)

1.2 The method may be used to analyze samples containing from 0.5 to 50 mg of chromium per liter.

1.3 This method may not be applicable to certain types of samples and extracts. Extracts contaning precipitate or cloudiness and those samples contaning solid material must be pre-treated by digestion using the Basic Digestion Procedure in Method 8.548

2. Summary of Method

2.1 Dissolved hexavalent chromium, in the absence of interfering amounts of substances such as molybdenum, vanadium, and mercury, may be determined colorimetrically by reaction with diphenylcarbazide in acid solution. A red-violet color of unknown composition is produced. The reaction is very sensitive, the absorbancy index per gram atom of chromium being about 40,000 at 540 nm. Addition of an excess of diphenylcarbazide yields the red-violet product and its absorbance is measured photometrically at 540 nm.

3. Sample Handling and Preservation

3.1 Since the stability of Cr(VI) in EP extracts is not completely understood at this time, the analysis should be carried out as soon as possible.

3.2 To retard the chemical activity of Cr(VI), the samples and extracts should be stored at 4°C until analyzed.

4. Interferences

4.1 The reaction with diphenylcarbazide is nearly specific for chromium. While hexavalent molybdenum and mercury salts will react to form color with the reagent, the intensities are much lower than that for chromium at the specified pH. Concentrations of molybdenum and mercury up to 200 mg/l can be tolerated. Vanadium interferes strongly but concentrations up to 10 times that of chromium will not cause trouble.

4.2 Iron in concentrations greater than 1 mg/l may produce a yellow color but the ferric ion color is not strong and no difficulty is normally encountered if the absorbance is measured photometrically at the appropriate wavelength.

5. Instrumental

5.1 Colorimetric equipment: One of the following is required:

1) Spectrophotometer, for use at 540 nm, providing a light path of 1 cm or longer.

2) Filter photometer, providing a light path of 1 cm or

longer and equipped with a greenish yellow filter having maximum transmittance near 540 nm.

5.2 Separatory funnels, 125-ml, Squibb form, with glass or teflon stopcock and stopper.

6. Reagents

6.1 Potassium Dichromate stock solution: Dissolve 141.4 mg of dried potassium dichromate, $K_2Cr_2O_7$ (analytical reagent grade), in deionized distilled water and dilute to 1 liter. [1 ml = 50 ug Cr]

6.2 Potassium Dichromate standard solution: Dilute 10.00 ml potassium dichromate stock solution to 100 ml. [1 ml = 5 ug Cr]

6.3 Sulfuric acid, 10% (v/v): Dilute 10 ml of distilled reagent grade or spectrograde quality sulfuric acid, H_2SO_4, to 100 ml with deionized distilled water.

6.4 Diphenylcarbazide solution: Dissolve 250 mg 1,5-diphenylcarbazide in 50 ml acetone. Store in a brown bottle. Discard when the solution becomes discolored.

6.5 Acetone: Analytical reagent grade. Avoid or redistill material that comes in containers with metal or metal-lined caps.

7. Procedure

7.1 Color development and measurement: Transfer 50 ml of the extract to be tested to a 100-ml volumetric flask. Add 2.0 ml diphenylcarbazide solution and mix. Add H_2SO_4 solution to give a pH of 2 ± 0.5, dilute to 100 ml with deionized distilled

water, and let stand 5 to 10 min for full color development. Transfer an appropriate portion of the solution to a 1-cm absorption cell and measure its absorbance at 540 nm. Use distilled water as reference. Correct the absorbance reading of the sample by subtracting the absorbance of a blank carried through the method (see also note below). From the corrected absorbance, determine the mg/l of chromium present by reference to the calibration curve.

NOTE: If the solution is turbid after dilution to 100 ml in 7.1 above, take an absorbance reading before addition of the carbazide reagent and correct the absorbance reading of the final colored solution by subtracting the absorbance measured previously.

7.2 Preparation of calibration curve: To compensate for possible slight losses of chromium during digestion or other operations of the analysis, treat the chromium standards by the same procedure as the sample. Accordingly, pipet measured volumes of chromium standard and stock solutions ranging from 2.00 to 20.0 ml, to give standards in the 0.5 to 50 mg/l Cr (VI) range when diluted to an appropriate volume, into 250-ml beakers or conical flasks.

7.3 Develop the color as for the samples, transfer a suitable portion of each colored solution to a 1-cm absorption cell, and measure the absorbance at 540 nm.

As reference, use distilled water. Correct the absorbance readings of the standards by substracting the absorbance of a reagent blank carried through the method. Construct a calibration curve by plotting corrected absorbance values against mg/l of chromium Cr (VI).

8. Verification

8.1 For every sample matrix analyzed verification is required to determine that neither a reducing condition nor chemical interference affecting color development is present. This must be accomplished by analyzing a second aliquot of the sample that has been spiked with Cr(VI). The amount of spike added should double the concentration found in the original aliquot. Under no circumstance should the increase be of less than 5 mg Cr(VI)/l. To verify the absence of an interference the spike recovery must be between 85% and 115%.

8.2 If the addition of the spike extends the concentration beyond the calibration curve the analysis solution should be diluted with blank solution, and the calculated results adjusted accordingly.

8.3 If the result of verification indicates a suppressive interference, the sample should be diluted and reanalyzed.

8.4 If, after dilution of the sample, the interference persists an alternate method (co-precipitation or chelation-extraction) should be used. Acidic extracts

which yield recoveries of < 85% should be retested to determine if the low spike recovery is due to the presence of residual reducing agent. This determination shall be made by first making an aliquot of the extract alkaline (pH 8.0 - 8.5) using 1N sodium hydroxide and then respiking and analyzing. If a spike recovey of 85-115% is obtained in the alkaline aliquot of an acidic extract that initially was found to contain < 5mg/l Cr VI one can conclude that the analytical method has been verified and the waste is not hazardous by reason of Cr VI EP toxicity.

8.5 If none of the analytical methods approved for this analysis yield valid results the sample is considered to exhibit the characteristic of EP Toxicity unless exempted according to the provisions of 40 CFR 261.4 (b)(6).

8.6 The method of standard addition will be used in accordance with the general instructions given in section 8.49.

Method 8.547
Chromium, Hexavalent
Chelation-Extraction/Atomic Absorption

1. Scope and Application

1.1 This method covers the determination of dissolved hexavalent chromium (Cr(VI)) in Extraction Procedure Toxicity Characteristic (EP) extracts. The method may also be applicable to certain domestic and industrial wastes provided that no interfering substances are present. (See 4.1.)

1.2 The method may be used to analyze samples containing from 10 to 250 ug of chromium per liter.

1.3 This method may not be applicable to certain types of samples and extracts. Extracts contaning precipitate or cloudiness and those samples contaning solid material must be pre-treated by digestion using the Basic Digestion Procedure in Method 8.548

2. Summary of the Method

2.1 This method is based on the chelation of hexavalent chromium with ammonium pyrrolidine dithiocarbamate (APDC) and extraction with methyl isobutyl ketone (MIBK). The extract is aspirated into the flame of the atomic absorption spectrophotometer.

3. Sample Handling and Preservation

3.1 Since the stability of Cr(VI) in EP extracts is not completely understood at this time, the chelation and extraction should be carried out as soon as

possible.

3.2 To retard the chemical activity of Cr(VI), the sample and extract should be stored at 4°C until analyzed.

4. Interferences

4.1 High concentrations of other reactive metals may interfere.

5. Instrumental Parameters (General)

5.1 Chromium hollow cathode lamp.

5.2 Wavelenth: 357.9 nm

5.3 Fuel: Acetylene

5.4 Oxidant: Air

5.5 Type of Flame: Fuel rich (adjust for organic solvent)

6. Reagents

6.1 Ammonium pyrrolidine dithiocarbamate (APDC) solution. Dissolve 1.0 g APDC in deionized distilled water and dilute to 100 ml. Prepare fresh daily.

6.2 Bromphenol blue indicator solution: Dissolve 0.1 g bromphenol blue in 100 ml 50-percent ethanol.

6.3 Potassium Dichromate standard solution I, [1.0 ml = 100 ug Cr]: Dissolve 0.2829 g pure, dried potassium dichromate $K_2Cr_2O_7$, in deionized distilled water and dilute to 100 ml.

6.4 Potassium Dichromate standard solution II, [1.0 ml = 10.0 ug Cr]: Dilute 100 ml chromium standard solution I to 1 liter with deionized distilled water.

6.5 Potassium Dichromate standard solution III, [1.0 ml = 0.10 ug Cr]: Dilute 10.0 ml chromium standard solution II to 1 liter ml with deionized distilled water.

6.6 Methyl isobutyl ketone (MIBK), Analytical reagent grade: Avoid or redistill material that comes in contact with metal or metal lined caps.

6.7 Sodium hydroxide solution 1 M: Dissolve 40 g sodium hydroxide, NaOH, ACS reagent grade, in deionized distilled water and dilute to 1 liter.

6.8 Sulfuric acid, 0.12 M: Slowly add 6.5 ml distilled reagent grade or spectrograde quality sulfuric acid, H_2SO_4, with deionized distilled water and dilute to 1 liter.

7. Procedure

7.1 Pipet a volume of extract containing less than 2.5 ug chromium (100 ml maximum) into a 200 ml volumetric flask, and adjust the volume to approximately 100 ml.

7.2 Prepare a blank and sufficient standards, and adjust the volume of each to approximately 100 ml.

7.3 Add 2 drops bromphenol blue indicator solution. (The pH adjustment to 2.4 may also be made with a pH meter instead of using an indicator.)

7.4 Adjust the pH by addition of 1 M NaOH solution dropwise until a blue color persists. Add 0.12 M H_2SO_4 dropwise until the blue color just disappears in both the standards and sample. The add 2.0 ml of

0.12 M H_2SO_4 in excess. The pH at this point should be 2.4

7.5 Add 5.0 ml APDC solution and mix. The pH should then be approximately 2.8.

7.6 Add 10.0 ml MIBK and shake vigorously for 3 minutes.

7.7 Allow the layers to separate and add deionized distilled water until the ketone layer is completely in the neck of the flask.

7.8 Aspirate the ketone layer and record the scale reading for each sample and standard against the blank. Repeat, and average the duplicate results.

8. Calculations

8.1 Determine the mg/l Cr(VI) in each sample from a plot of scale readings of standards. A working curve must be prepared with each set of samples.

9. Verification

9.1 For every sample matrix analyzed verification is required to determine that neither a reducing condition nor chemical interference affecting chelation is present. This must be accomplished by analyzing a second aliquot of the sample that has been spiked with Cr(VI). The amount of spike added should double the concentration found in the original aliquot. Under no circumstance should the increase be of less than 30 ug Cr(VI)/l. To verify the absence of an interference the spike recovery must be between 85% and 115%.

9.2 If the addition of the spike extends the concentration beyond the calibration curve, the analysis solution should be diluted with blank solution and the calculated results adjusted accordingly.

9.3 If the result of verification indicates a suppressive interference, the sample should be diluted and reanalyzed.

9.4 If, after dilution of the sample, the interference persists an alternate method (coprecipitation or colorometric) should be used. Acidic extracts which yield recoveries of < 85% should be retested to determine if the low spike recovery is due to the presence of residual reducing agent. This determination shall be made by first making and aliquot of the extract alkaline (pH 8.0 - 8.5) using 1N sodium hydroxide and then respiking and analyzing. If a spike recovery of 85-115% is obtained in the alkaline aliquot of an acidic extract that initially was found to contain < 5mg/l Cr VI, one can conclude that the analytical method has been verified and the waste is not hazardous by reason of Cr VI EP toxicity.

9.5 If none of the analytical methods approved for this analysis yield valid results, the sample is considered to exhibit the characteristic of EP Toxicity unless exempted according to the provisions of 40 CFR 261.4 (b)(6).

9.6 The method of standard addition shall be used in accordance with the general instructions given in section 8.49.